

Fine Powder PTFE

PROCESSING GUIDE

INTRODUCTION

Fine Powder (FP) PTFE (polytetrafluoroethylene) is a high molecular weight, fluoropolymer resin. Because of its high melt viscosity, PTFE cannot be melt extruded or formed like most other polymers. PTFE Fine Powders have specifically been developed such that thin walled and other fine objects can be formed by a unique cold extrusion process called "Paste extrusion". PTFE Fine Powders are a very porous material with Specific Surface Areas as high as 12 square meters per gram. Therefore it is possible to add a volatile lubricant to these powders, which distributes itself uniformly within the powder. This mixture becomes in fact the "paste" = powder + lubricant. Before extrusion this paste is typically compressed, to remove as much air as possible, into a cylindrical perform or "candle". The perform is subsequently placed in a special constant rate ram extruder and the PTFE is extruded into the desired shape. Such extrusions are typically performed at powder temperatures of 35 - 50 °C. During extrusion many of the particles change into fibrils, which give strength to the extrudate. Subsequently the extruded parts are dried for lubricant removal and then sintered at 360 to 400°C, when melting occurs and the objects become uniform and air free.

Typical products made from PTFE Fine Powders are tubes, wires, tapes and membranes, but there are other applications also. Typical end-uses are high quality hoses and wires for the aerospace and automotive industries, pipe liners and transfer hoses for corrosive materials and solvents in the chemical industry, membranes for apparel and filtration, etc. Depending on the type of processing equipment, the products desired or the final properties, different grades of PTFE Fine Powders are required. GFL offers a complete range of different INOFLON®FP types to meet the broad needs of various industries.

USEFUL PROPERTIES OF PTFE

PTFE has a very wide array of unique physical, chemical and mechanical properties that make it the material of choice for the most demanding applications.

- Extremely low coefficient of friction
- Non-stick
- Low dielectric constant and loss factor
- Broad range of service temperature (-260°C to +260°C)
- Weather resistance /UV resistance/ hydrophobic
- Excellent chemical and solvent resistance
- Compliance for Food Contact (FDA, other)

STORAGE

INOFLON® FP PTFE is susceptible to shear damage, particularly above its transition point 19°C (66.2°F). Handling and transportation of the containers could easily subject the powder to sufficient shear to spoil it if the resin temperature is above transition point. To ensure that the resin does not fibrillate, it should be cooled below its transition temperature prior to handling and transportation. A typical commercial container (20–30 kg) should be cooled for 24–48 hours to <15°C (59°F) to assure temperature uniformity throughout the container. Specially designed shallow cylindrical drums are used to minimize lump formation, compaction, and shearing of the resin.

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To prevent moisture contamination, the drum must not be opened where the ambient dew point is above the temperature of resin to avoid immediate condensation on the resin. Storage and handling facilities should be clean. Good housekeeping and careful handling are essential.

SAFETY IN USE

PTFE is very inert and therefore quite safe in use. Nevertheless some basic housekeeping rules are needed. These rules are listed in labels on the drums. We strongly recommend being familiar with the contents of the brochure "Safe Handling of Fluoropolymers" which can be downloaded from www.fluoropolymers.org

FINE POWDER GRADE SELECTION

An important concept in the Paste Extrusion of PTFE Fine Powders is the so-called Reduction Ratio (RR). Reduction Ratio is the ratio of the cross-sectional area of the PTFE in the barrel of the extruder to the cross sectional area of the PTFE in the extrudate. If for example a thin wire is extruded from a large diameter extruder than the RR is also quite large. Important is then to have products that develop a lower extrusion pressure under these conditions. On the contrary, if a large diameter pipelining tube is extruded than the RR will be very low, because there are practical limits as to the size and performance of extruders. In a very broad manner it can be said that the great majority of RR's lie between 10:1 and 4000:1. Choice of a material often depends on both equipment available, the dimensions of the product being extruded and final properties. The exact same end product can be produced at different RR, depending on the size of extruder used.

The most important property that determines the backpressure is the amount of fibrillation. PTFE Fine Powder is very large surface area polymer (greater than 10 m² /g), which consists of agglomerates of a large number of small particles – of approximate average size 200 to 300 nanometers. Under high shear forces many of these particle will undergo a transformation into small fibrils. This fibrillation is still present in some final product such as Thread Sealant Tape which has been mono-axially stretched, or in membranes for filtration which have been bi-axially stretched. High fibrillation can be found in low RR products, while low is present in the high RR types.

GFL has created and offers a range of Fine Products that are suitable for these various needs. While RR is a very important parameter for the extrusion of PTFE Fine Powder, manufacturers also strive to impart to their end products the best suitable final properties. These can range from stretch ability, low permeation, good flex-life, and transparency to physical properties and other. It is important then to select the best product that balances best both the extrusion needs and the end-use requirements. Do not hesitate to contact GFL in order to discuss the best product options for the application.

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INOFロン® FINE POWDER PRODUCTS AND APPLICATIONS

GN 7001 - For low Reduction Ratio (RR) applications. Typical end uses are high performance tapes, gaskets, yarns. Good stretching properties.

GN 7003 - For low RR applications. Typical end-uses are tapes, yarns, fibres, dental flosses and membranes. Good stretching properties.

GN 7040 - For low and medium RR applications. Typical end uses are sealant thread seal tapes, thick wall tubes, pipe liners, sealants, etc. Good stretching properties.

GN 7045 - For low to medium RR applications. Typical end uses are tubings.

GN 7300 - For high RR applications. Typical end uses are spaghetti tubes, wire & cable insulations, thin wall tubes.

GN 7500 - for very high RR application. Typical end used are thin wall tubing, spaghetti tubing, small diameter tubing, wire and cable insulations."

MGN 7045 - Modified polymer. For low to medium RR applications. Well suited for high-end tubes for demanding applications. Excellent flex-life, low permeability, transparency. Also suitable for some electrical grade tape applications.

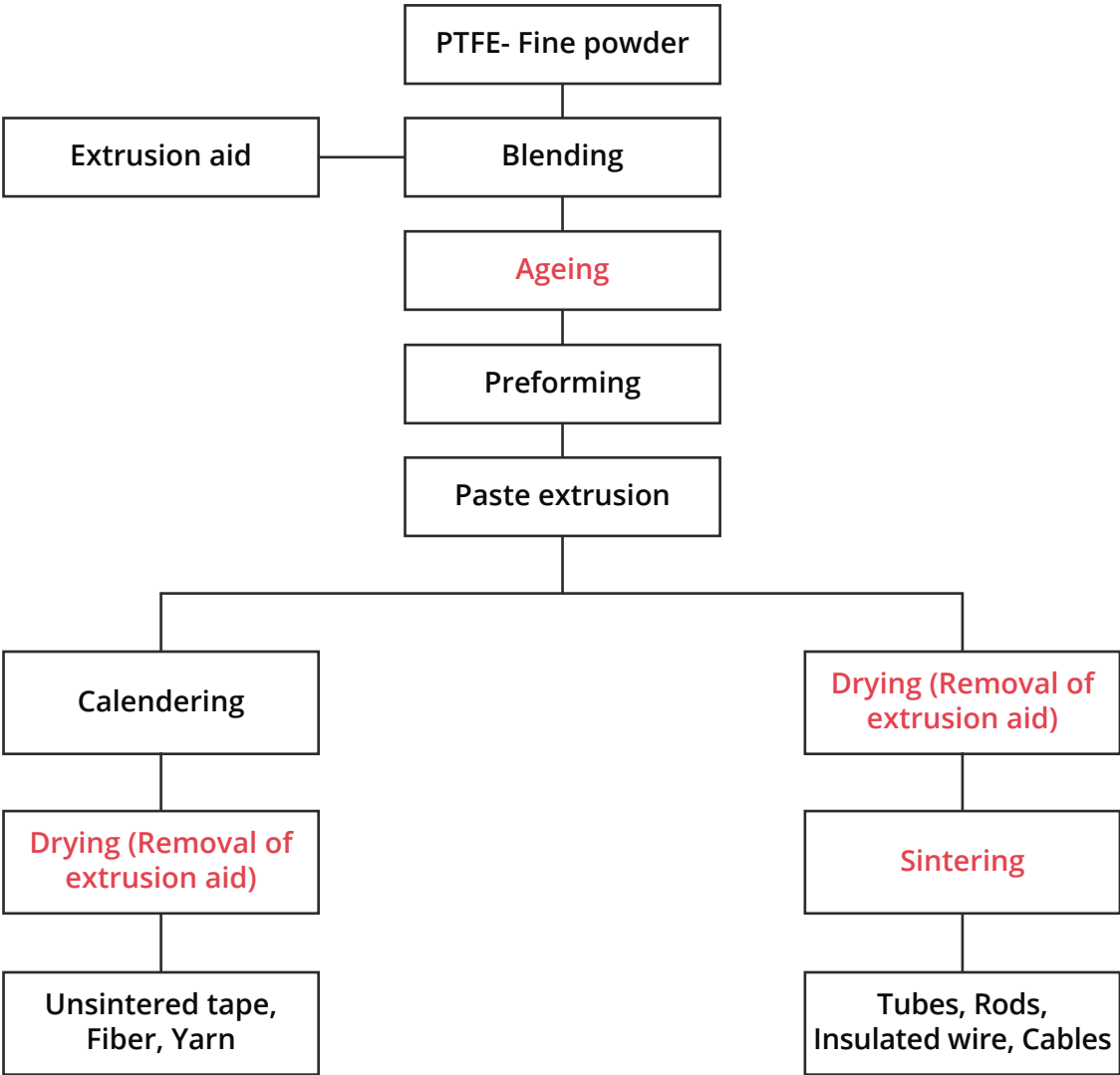
MGN 7055 - Modified polymer. For medium RR applications. For high end tubes with excellent flex life, low permeability & transparency.

MGN 7065 - Modified polymer. For high RR applications. For high end tubes with excellent flex life, low permeability & transparency.

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TYPICAL PROCESSING STEPS OF FINE POWDER PTFE

- 1.Preparation – Sieving
- 2.Mixing in of extrusion aid – Lubrication
- 3.Ageing and warming up of mixture
- 4.Preforming
- 5.Batch extrusion
- 6.Calendering – For tape applications
- 7.Drying – Lubricant removal
- 8.Stretching – For tape, gasket material, fibers, yarns
- 9.Sintering – Melting – Certain products such as tapes do not undergo this melting step
- 10.Post-finishing steps – Modifications on the basic extruded shapes such as convolution of tube's



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EXTRUSION OF UNSINTERED ROD AND THREAD SEAL TAPE

SIEVING

Fine powder can be compacted to a small extent during transportation and storage, even when refrigerated and handled with care, thereby creating lumps. Sifting the cold resin through coarse wire mesh will help break up these lumps. The size of the sieve should be 3-5 mm. Since PTFE Fine Powder is shear sensitive care must be taken in handling the powder, therefore the resin should not be scooped out of the container but gently poured over the sieve

MIXING OF EXTRUSION AID (LUBRICANT)

In order to extrude PTFE fine powder, it must be first uniformly mixed with a lubricant. This extrusion aid lowers both the internal and external friction present during extrusion. This lubricant needs to be compatible with PTFE and be able to wet it well, and have a relative low boiling point such that it can be easily removed after extrusion. There are two basic types of lubricant, light or low boiling and heavy or higher boiling, depending on the application. For the manufacture of tapes and beads a heavier lubricant is required, such that it does not evaporate during post extrusion operations such as forming. A typical lubricant for these end-uses is Isopar M (Exxon) with a boiling range of about 200 - 250°C. For other end-uses such as tubes and wires a lighter lubricant is typically used, with boiling ranges that may vary from 110 to 160°C. Examples of such lubricants are respectively Isopar E ,G and H.

The amount of the lubricant in the compound depends on the type of the product, equipment design, and the desired extrusion pressure.

Its content should be as low as possible such that drying is easy and fast, but not so low that the extrusion pressure would be excessively high. The range of lubricant content is generally 15–20% based on the total weight of the compound.

Blending the lubricant with PTFE, must be conducted in a clean enclosed area where the temperature is below the resin transition temperature (19°C). Bottle or jar method are commonly used to blend the lubricant with resin. The bottle or jar method (see Fig.) requires a wide mouth stainless steel or glass container for easy (low shear) powder loading. Plastic containers can be used, but care should be taken to use polymers with low permeation, such as PET, otherwise lubricant may be lost during ageing and storage.

The jars must be sealed tightly to prevent the loss of the lubricant by evaporation. Large industrial mixers such as Twin Shell can also be used. The following steps should be taken to prepare the paste extrusion compound in jars or bottles.

1. Weigh the powder after screening and carefully load in to the container.
2. Create a cavity by giving the bottle a rapid twist.
3. Pour the lubricant into the cavity in the middle of the powder.
4. Close the lid and place the bottle on rollers (15 rpm) for 20–30 minutes.
5. Small lumps can be broken up if need be by resieving and the lubricated powder rerolled for 3–5 minutes.
6. Empty the blender and store the lubricated resin in a jar or the original drum and make sure the lid is tightly sealed.
7. Allow the blend to age for at least 12 hours at 30°C to allow complete diffusion of the lubricant into the the polymer particles and to bring the powder to come up to required extrusion temperature

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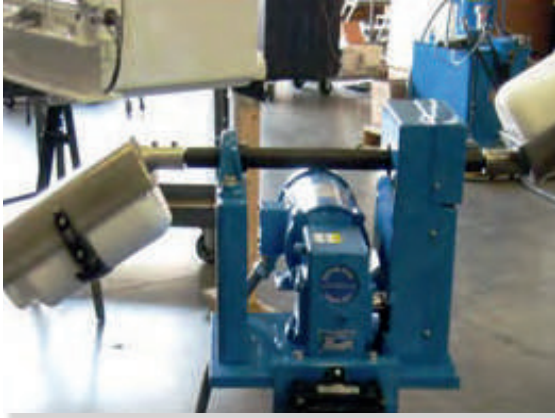


Fig- Typical PTFE/lubricant jar blender



Fig- Industrial type V blender

ADDITION OF PIGMENT (OPTIONAL)

Pigments in dry form or as a dispersion can be added to PTFE to color the for example insulation of wires.

Pigment should have the following characteristics.

1. It should not be affect the electrical properties of PTFE
2. It should be capable to withstand the high sintering temperatures.

There are two technique used to mix the pigment with resin.

Method 1

Pigment loading of any type in the final insulation should not exceed 1% because of its detrimental effect on the dielectric properties.

Inorganic pigments should be selected for coloring PTFE because almost all organic pigments will completely degrade at the sintering temperatures of the polymer. Following steps are used to mix the pigment in resin.

- Pigment should be first screened with 250 mesh sieve before being added to the dry PTFE.
- After adding the pigment to the resin, the container should be rolled or tumbled for 5–10 minutes to mix the pigment throughout the dry resin.

Method 2

- Pigment first added to the lubricant which is to be used.
- Then mixture (pre-dispersion) of pigment and lubricant is then added to the resin.
- Tumble the container for 5 to 10 minutes to mix the pigment throughout the dry resin.

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PREFORMING

Prior to extrusion it is necessary to remove as much air as possible from the PTFE/Lubricant paste, otherwise defects can occur in the extrudate. Furthermore, in order to maximize the batch material capacity, increasing the density of the paste is also desirable. The mixture is therefore pressed into cylindrical shapes, so-called preforms or billets. For the extrusion of tubes and wires preforms are made in a cylinder equipped with a mandrel and a piston as shown in Fig(1). The mandrel is positioned in the center of the cylinder and the resin is charged in the annular space. In the production of beading such as those needed for tape manufacturing, there is no mandrel. The aged lubricated resin without lumps is loaded into the preform cylinder and is evenly distributed around the core mandrel press until the volume of powder is reduced to approximately 1/3 of its original volume to the preform. Pressure used for preforming should be 0.5 to 1.5 MPa, and pressing speed of 50 mm/min is recommended.

The preform is quite weak and can easily break or deform, therefore it requires care during removal from the cylinder. The preform should be loaded in the extruder immediately after removal, since lubricant can quickly evaporate from the surface of the preform. Preforms can also be stored for longer periods of time if kept in tight containers

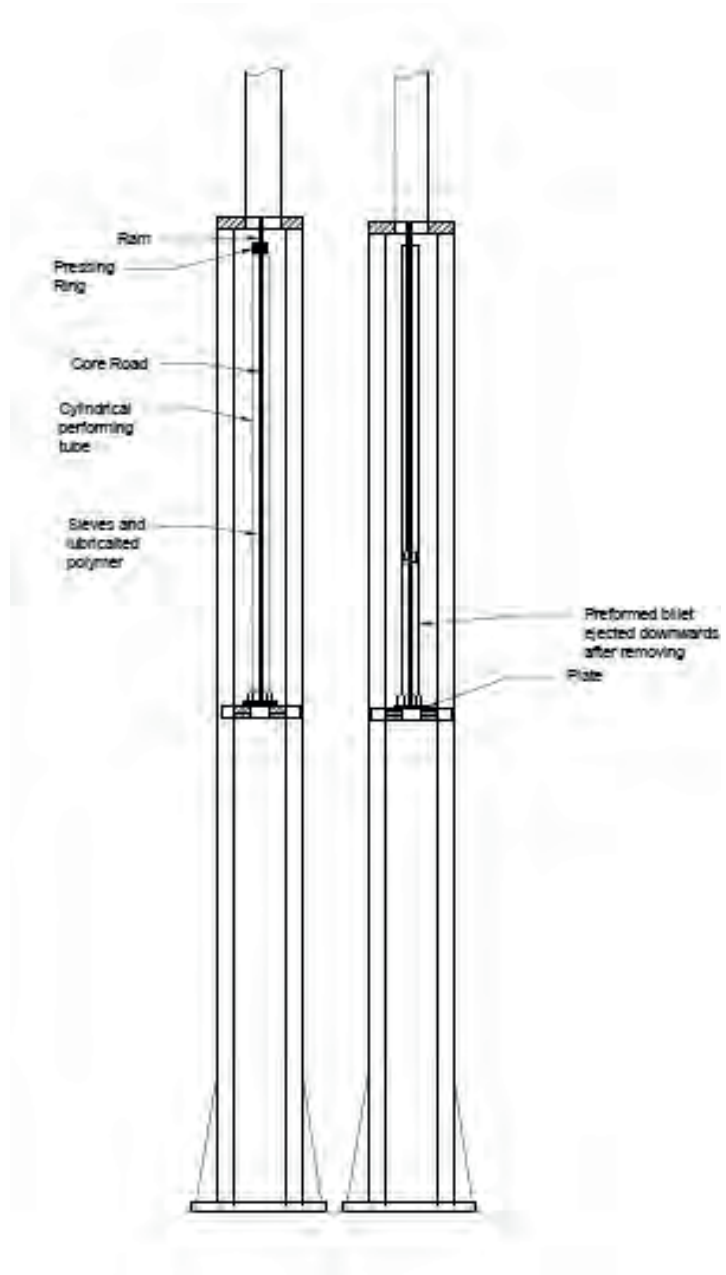


Fig (1) - Schematic of a preform press

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EXTRUSION

After performing the Fine Powder paste in now ready for extrusion. Most extrusions are either vertical or horizontal, and examples of such extruders are shown in figs (2),(3). In rare cases extrusions can be performed at a certain angle from the horizon. Most extrusions are horizontal because of ease of accessibility to the equipment. Both horizontal and vertical configurations have advantages and disadvantages, such that the choice requires careful consideration. These considerations include the handle ability of the extrudate after exiting the extruder, factory space needed or available, accessibility and more. Extrusion of PTFE Fine Powder is a batch process. With a few exceptions, this means that the length of tubes or wires is limited to the amount that can be fed to one batch extrusion. Extrusion of tubes and wires requires the presence of a mandrel in the barrel, which is attached to the back part. For wires & cables, a conductor is fed through this mandrel for tape manufacturing a solid bead is extruded, for subsequent calendaring and stretching.

Extruders are typically powered by sophisticated hydraulic systems, capable of maintaining an accurate and constant rate of extrusion piston travel. Some extruders are driven by mechanical systems such as motor driven screw types.

Extruder diameters may be as small as a few inches up various tens of inches, depending on the application. The stroke can be as large as 108", and the force can go up to as high as 100 ton. The tips of the extruder can be changed such that the desired dimensions of the final product can be achieved.

As previously mentioned, the PTFE should be maintained at a temperature above its room transition point. Extrusions are typically performed at temperatures of 30 to 50 °C.



Fig (2)- Vertical Extruder

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Fig (3)- A Jennings International Horizontal Extruder.

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DRYING AND SINTERING

After extrusion of tubes or wires the lubricant is removed by heating to typically 200 to 250°C. It is important to regulate the system such that solvent evaporation is gentle in a way that the extruded product is not damaged. Care must also be maintained that often flammable extrusion aids are handled in the proper way in this step. Temperature profiles in the drying oven depend on thickness and diameter of product, extrusion speed and type of lubricant used.

In the production of tapes, the extruded bead is first calendared and only then follows the drying step.

Drying can be done in a continuous fashion, coupled to the extrusion and the subsequent sintering step. Batch drying is less common; it can take place in drying oven or ambient enclosures with good ventilation.

Sintering often complements the drying step and occurs in similar ovens. It is very important that the products reaching the sintering zone be absolutely free of lubricant. In these ovens all of the polymer should be taken above its melting point, which is approximately 345°C. In view of the speed and thermal insulating properties of PTFE, it is generally necessary to set the ovens to well above this temperature. It is critical to make sure the actual polymer temperature does not go above 400°C, otherwise substantial degradation may start occurring. As the products exit the sintering ovens and the polymer goes from a molten state, to a frozen one, the cooling rate may for some types be important. Because low crystallinity is generally desirable, this can be achieved when necessary by quenching the products in cold water as they exit the ovens.

POST-EXTRUSION FINISHING STEPS – CALENDERING & STRETCHING

CALENDERING

Solid PTFE beads produced in extrusion can be further transformed into useful final products. The bead itself can be stretched in order to include more air, and thereby increase its elasticity. Such products make excellent gasketing materials.

The bead can also be fed warm into a calendar and flattened into a tape of the desired thickness. Calendaring can be done by in a twin roll calendar with adjustable gap in the thickness range. The width and diameter of the rolls is determined by the width of the tape to be made. Larger diameter rolls should be selected for wider tapes.

For example, to calendar a bead into a 100–200 mm wide tape, the calendar diameter should be 300 mm and its width about 300 mm. The rolls should be equipped with internal water or oil heating capable of providing a temperature of 50–80°C to facilitate the calendaring of the bead. A continuous drive should provide roll speed in the range of 1.5–40 m/min. More than one set of calendaring rolls can be used to reach the desired thickness. In the case of multiple calendars, only the gap in the last calendar would have to be precisely controlled.

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DRYING OF TAPE

A variety of oven designs have been satisfactorily used in the removal of lubricant. They include internally heated flat plates, heated drum, resistance heaters and infrared lamps. The ovens must be thoroughly exhausted to assure that hydrocarbon concentration is well below its lower flammability limit. Fig (4) Depicts a typical convection drying oven in conjunction with a stretching step. The temperature settings depend on the volatility of the solvent.

STRETCHING

Stretching is necessary to add more air to the tape produced. It increases the porosity of the product, and its density is lowered. Stretching is generally performed at high temperatures but below melting temperature of PTFE (342°C) , after the drying the calendared tape.

Stretching can be done mono-axially in order to yield products such as gasketing and thread seal tape, or bi-axially for membranes. Control of uniform pore size is essential for these applications.

Stretching is possible due to the fibril structure of the extruded bead. New fibrils may be formed in this step. The fibril can be oriented as desired in the stretching step.

For these applications PTFE Fine Powders with good fibrillation and stretching properties are required.

After stretching the product can be cut or slit to the required dimensions.

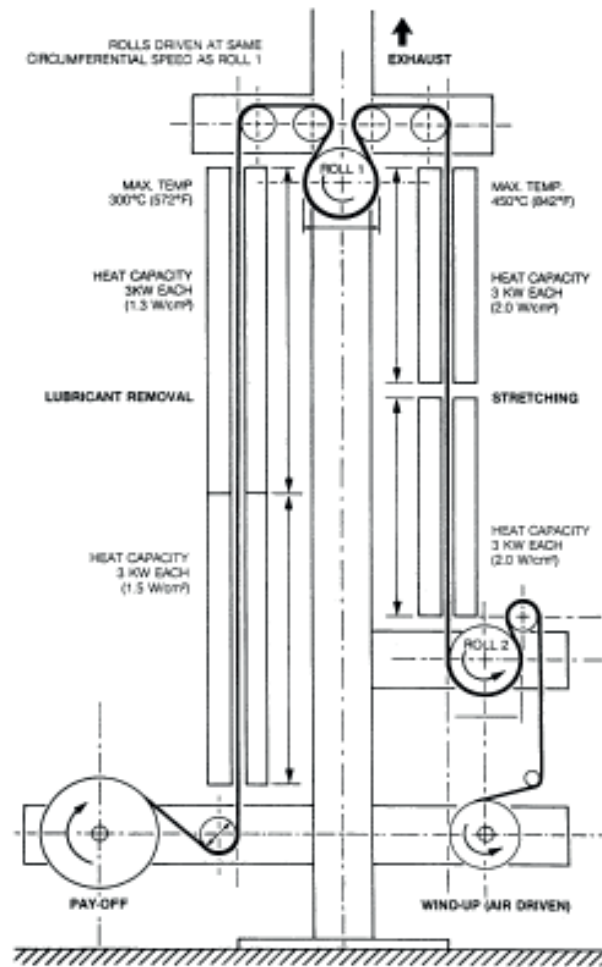


Fig (4)- Drying oven for removing lubricant.

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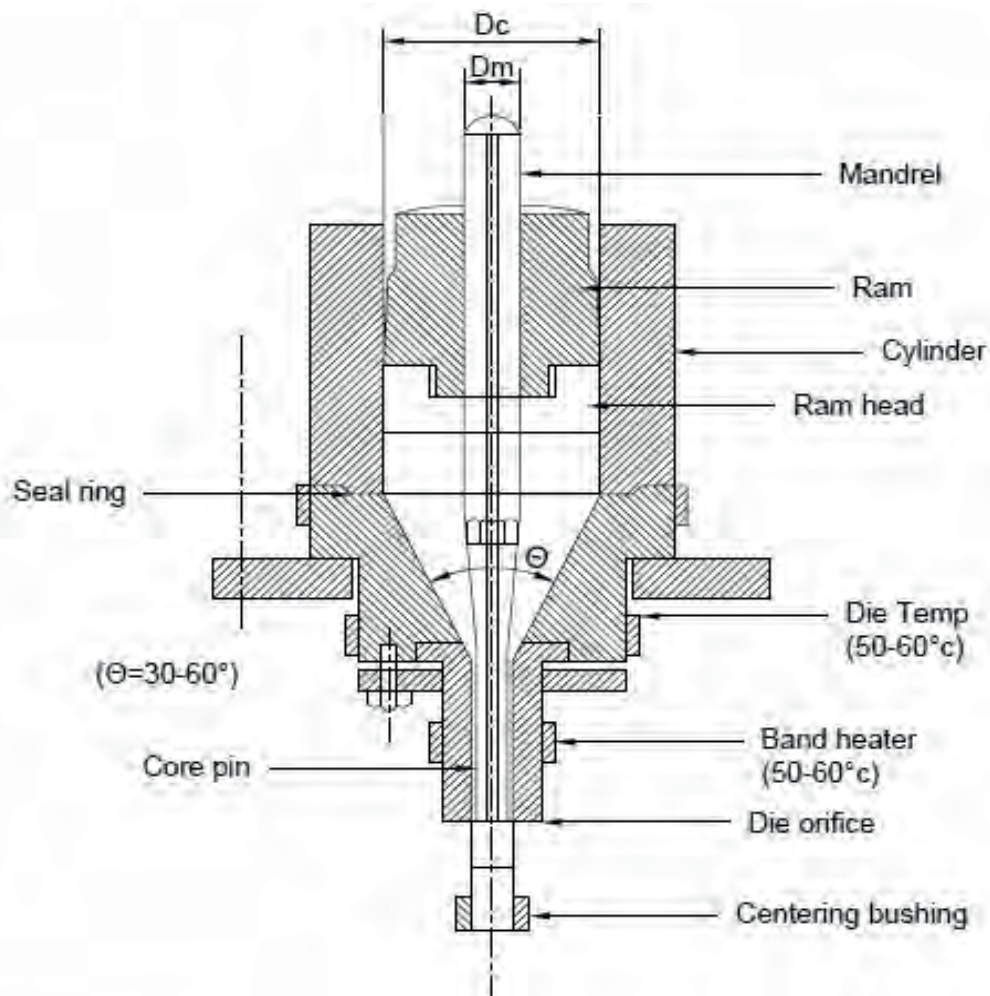
EXTRUSION OF SINTERED TUBES

Resin lubrication, pigmenting, and preforming should be done similarly to preparations described for type of lubrication may change, but the basic method mixing, blending, handling of resin remain the same. Any differences in preparations have been pointed out wherever necessary.

The majority of tubes made from PTFE by paste extrusion have fairly thin walls (<3 mm) and are produced in a wide size range from a fraction of millimeter to several centimeters in diameter for applications ranging from fluid transfer in healthcare to fuel and hydraulic transfer in jet engines.

Two type of extrusion technique are used to manufacturing the tube, horizontal extrusion and vertical extrusion.

The basic tube extrusion equipment shown in fig(5) the extruder consist the extrusion barrel and die. A wide range of reduction ratio (cross sectional area of interior passage of barrel divided by cross sectional area of extrusion orifice) may upto 2000:1 used. Either a hydraulic or a screw-type ram driving mechanism is suitable. Hydraulic-type driving mechanism is most often used in large-scale extruders. The preform fibrillates in the die under ram pressure and forms an extrudate, which should have the right thickness and smoothness. The design of the die plays a key role not only in the property & quality but also on the extrusion pressure. Die angle depends on reduction ratio, 20-40° angle are suitable, die land length 3 to 10 times longer than it's diameter. Extrusion moulding is done at room temperature 21-25°C where as die heated at 30 to 50°C.



Fig(5)- Schematic of tube extrusion die.

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DRYING AND SINTERING

After extruded the tube lubricant must be removed by heating and it is done by the inline oven mounted, vertically underneath the extruder, Temperature of drying zone should keep 150 to 250°C for complete removal of lubricant and the temperature is adjusted by oven heating source. If the temperature of heating zone is too high lubricant may boil and cause split and blisters in the extruded.

It is not easy to generalize the respective length of drying and sintering zone it depends on the thickness of the product, the thicker the section the greater is the required proportion drying oven to sintering oven.

After the complete removal of the lubricant of extruded in drying section temperature is raised 370-390°C for sintered product and the temperature of sintering zone depends on various factors such as thickness and diameter of the product, extrusion speed etc.

Sintering oven should be capable of heating the extrudate to well above the PTFE melting point at about 345°C

Cooling step must be slow because properties of product like permeability, crystallinity, flexibility depends on cooling rate of PTFE.

EXTRUSION OF INSULATION WIRE WITH PTFE

EXTRUSION EQUIPMENT AND PROCESS

Figure (6) shows a schematic of a paste extrusion line for wire insulation. The wire is passed through the paste extruder where it is coated with PTFE while moving through the die. The ram extruder for this process is a special unit and can be either horizontally or vertically oriented, which refers to the direction of the ram movement.

The extruder consists of a barrel, and a hydraulic or screw-driven ram, a die, a mandrel etc. the cylinder with inside diameter of 25-100 mm is generally used, but it can be changed as design of product.

The preform is forced through the die by the force of the ram. It is important to be able to control the speed of the ram on a continuous scale and keep it literally constant at a set speed. The uniformity of the thickness of the coating is critically dependent on the constancy of the ram speed. The speed of the ram is continuously adjustable usually over range of 0-50 mm/min. The hydraulic or mechanical drive system must be capable of supplying the force necessary to extrude the preform. Ram pressure capability up to 150 MPa may be required for extrusion at high reduction ratios.

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The design of the die plays a key role not only in the property and quality of the coating but the extrusion pressure. The angle of the die wall (cone) affects the surface smoothness of the extrudate. The range of the angle is 15–60° but an angle of 20° for thin coatings on fine conductors and an angle of 30° for thicker coatings have been recommended. The conductor (wire) emerges from the guide tube at a tip which guides it through the segment called the die land. The location of the tip is critical to the insulated wire quality because it affects how the wire and PTFE are brought together.

Adjusting the position of the tip can control coating thickness, stripability of the PTFE (tightness) and the number of flaws.

If the clearance is too small, the tip constrains the movement of the paste and extrusion pressure rises. Small clearances also reduce the tightness of the insulation around the conductor. If the clearance is too large the wire tension increases because of the drag of the paste on the conductor. It also elevates the tightness of the insulation and may lead to cracking. Larger clearance will allow the paste to extrude backwards into the tube. Apart from tip clearance, properties of coated wire influenced by die angle, dia diameter and die land length. The extrusion speed is determined by the material's shear sensitivity and the conditions in the down stream drying & sintering ovens

DRYING AND SINTERING

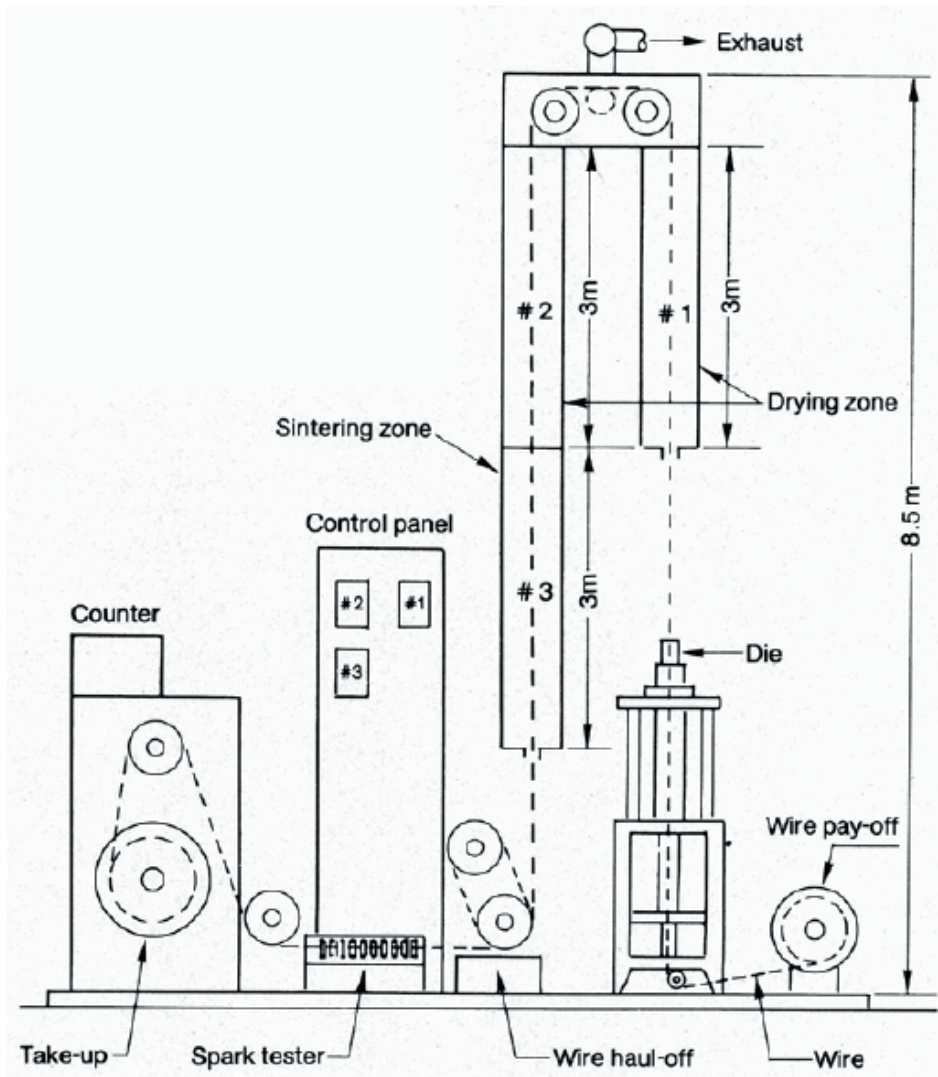
Lubricant must be removed prior to sintering. The coating may be crack, if it contains a large amount of the lubricant and reaches the sintering zone. Any remaining lubricant will degrade at the sintering temperatures and leave a colored residue.

The exact temperature depends on the speed of the wire and lubricant type. The wire enters the sintering zone immediately after it leaves the drying oven

It still lacks strength because of its porous unsintered structure. PTFE undergoes coalescence and void elimination during sintering. The polymer must be heated to, at least, its melting point of 342°C for a brief period of time for melting to actually occur. The oven temperatures are typically set at 350– 450°C, based on a number of variables including the speed of the wire and thickness of the coating. Heat transfer to the polymer accelerates at these temperatures, but care must be taken to prevent exposure of PTFE to temperatures above 390°C where degradation begins to speed up.

The sintering ovens should be equipped with an exhaust system to remove the toxic by-products in case of PTFE degradation. In cooling process The exiting wire insulation is in the molten state, solidifying upon contact with the ambient air the wire is usually allowed to cool by natural convection in the ambient air blowers can be installed to move the warm air away from the area.

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Fig(6)- Extrusion Molding of Electric Wire Insulating Extrusion Process PTFE Fine Powder ("Jennings" Electric Wire Insulating System (Hydraulic.)

The information in this processing guideline is believed to be correct to the best of our knowledge and information. It is subject to revision depending on the additional knowledge gained by us with the time. The information provided in this processing guideline should be treated as guidance and not to be considered as warranties or quality specification.

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